

SEPARATION OF *d-neo*BORNYLAMINE
FROM *d*-BORNYLAMINE

BY

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Separation of d-Neobornylamine from d-Bornylamine. By
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It has been shown by Forster (J., 1898, 73, 390) that reduction of *d*-camphoroxime with sodium in boiling amyl alcohol yields a mixture of *cis*- and *trans*-isomerides, distinguished as *d*-bornylamine and *d*-neobornylamine, respectively. The isolation of *d*-neobornylamine from this mixture has presented considerable difficulty (compare Forster and Hart-Smith, J., 1900, 77, 1152; Pope and Read, J., 1910, 97, 987; 1913, 103, 454). Separation of the two amines can be effected in the following way.

A mixture of the two amine hydrochlorides (223 g.), resulting from the reduction of *d*-camphoroxime (290 g.), on recrystallisation from water gave a crop of crystals (77.3 g.). An ethereal solution of the bases from the crystals was shaken consecutively with 500

and 300 c.c. of dilute hydrochloric acid (approx. 0.6*N*), giving solutions 1 and 2. A similar solution of the bases from the mother-liquor was shaken with three quantities of 500 c.c. of the dilute acid, giving solutions 3, 4, and 5. These five preparations were evaporated to dryness, giving the following products :

No.	Weight.	$[\alpha]_D$ in dry alcohol ($c = 4.4$).	Bornylamine hydrochloride. Calc.	Neobornylamine hydrochloride. Calc.
1	49.8 g.	+19.1°	46.9 g.	2.9 g.
2	27.5	+12.9	23.6	3.9
3	50.85	+ 3.7	37.15	13.7
4	50.35	— 6.2	29.9	20.45
5	44.5	—22.6	16.6	27.9

Pure *d*-bornylamine hydrochloride was easily obtained from the dextrorotatory fractions by recrystallisation from water as shown by Forster (*loc. cit.*) and Frankland and Barrow (J., 1909, 95, 2017). The specimen giving the highest rotation had $[\alpha]_D^{20} + 23.3^\circ$ ($c = 4.4$ in dry alcohol), the previous highest value recorded being $+ 22.7^\circ$ (Forster, *loc. cit.*). The ethereal solutions of the bases from the lævorotatory fractions were shaken with small quantities of dilute hydrochloric acid until the $[\alpha]_D$ of the hydrochloride of the base remaining in the ethereal solution became constant. *d*-Neobornylamine hydrochloride having $[\alpha]_D^{20} - 49.4^\circ$ ($c = 4.4$ in dry alcohol) was thus obtained, the previous highest value recorded being $- 44.2^\circ$ (Forster and Hart-Smith, *loc. cit.*).—WELLCOME CHEMICAL RESEARCH LABORATORIES. [*Received, March 19th, 1927.*]

